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## Structure Reports

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# (E)-2-[(4-Chloro-1,3-dimethyl-1H-pyrazol-5-yl)methyleneamino]benzamide

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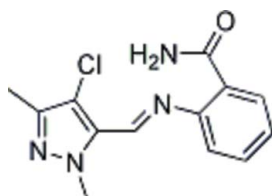
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 Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.035;  $wR$  factor = 0.098; data-to-parameter ratio = 13.6.

In the title compound,  $\text{C}_{13}\text{H}_{13}\text{ClN}_4\text{O}$ , the dihedral angle between the aromatic rings is  $33.47(9)^\circ$  and an intramolecular  $\text{N}-\text{H}\cdots\text{N}$  hydrogen bond generates an  $S(6)$  ring. In the crystal, inversion dimers linked by pairs of  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds occur, resulting in  $R_2^2(8)$  loops.

## Related literature

 For catalytic studies on a related compound, see: Chen *et al.* (2008).


## Experimental

## Crystal data

 $\text{C}_{13}\text{H}_{13}\text{ClN}_4\text{O}$ 
 $M_r = 276.72$ 

 Monoclinic,  $C2/c$   
 $a = 22.046(3)$  Å  
 $b = 8.6785(10)$  Å  
 $c = 14.1137(16)$  Å  
 $\beta = 96.005(2)^\circ$   
 $V = 2685.5(5)$  Å<sup>3</sup>
 $Z = 8$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.28$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.32 \times 0.30 \times 0.28$  mm

## Data collection

 Bruker SMART CCD diffractometer  
 Absorption correction: multi-scan (*SADABS*; Bruker, 2001)  
 $T_{\min} = 0.725$ ,  $T_{\max} = 1.000$ 

 6634 measured reflections  
 2375 independent reflections  
 2098 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.018$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.035$   
 $wR(F^2) = 0.098$   
 $S = 1.06$   
 2375 reflections

 175 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.17$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.19$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N4}-\text{H4B}\cdots\text{N3}$	0.86	2.07	2.749 (2)	136
$\text{N4}-\text{H4A}\cdots\text{O1}^i$	0.86	2.10	2.930 (2)	161

 Symmetry code: (i)  $-x + \frac{1}{2}, -y + \frac{1}{2}, -z$ .

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5252).

## References

- Bruker (2001). *SMART, SAINT and SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Chen, J., Su, W., Wu, H., Liu, M. & Jin, C. (2008). *Catal. Commun.* **9**, 785–788.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

## supporting information

*Acta Cryst.* (2009). E65, o3263 [doi:10.1107/S1600536809050685]

## (*E*)-2-[(4-Chloro-1,3-dimethyl-1*H*-pyrazol-5-yl)methyleneamino]benzamide

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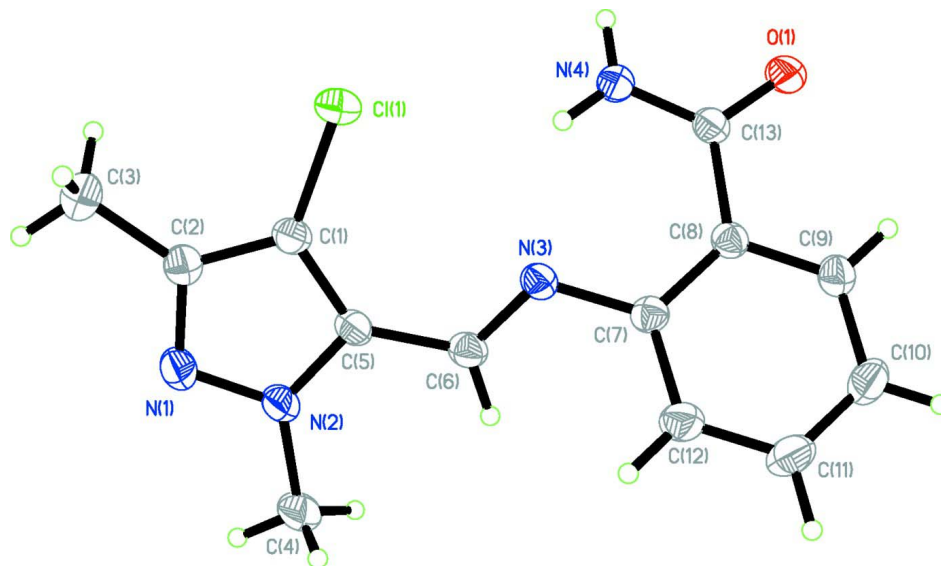
### S1. Experimental

The title compound was synthesized by the reaction of 4-chloro-1,3-dimethyl-1*H*-pyrazole-5- carbaldehyde with 2-aminobenzamide in 1,2-dichloroethane. The crude product was purified by silica-gel column chromatography and then grown from methylene chloride to afford colourless blocks of (I).

Anal. Calcd for C<sub>13</sub>H<sub>13</sub>N<sub>4</sub>O<sub>1</sub>: C, 56.42; H, 4.74; N, 20.25; O, 5.78. Found: C, 56.57; H, 4.64; N, 20.66; O, 5.25. <sup>1</sup>H NMR(DMSO): 2.21(s,3H, CH<sub>3</sub>), 4.12 (s,3H, N—CH<sub>3</sub>), 7.33(s, 2H, CONH<sub>2</sub>), 7.50–7.61 (m, 2H, Ph), 7.89 (t, 1H, Ph—H), 8.26 (s, 1H, Ph—H), 8.46 (s, 1H, CH=N).

### S2. Refinement

All H atoms were visible in difference maps; they were finally placed in geometrically calculated positions, with C—H = 0.93–0.96 Å and N—H = 0.86 Å, and refined as riding with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$  or  $1.5U_{\text{eq}}(\text{methyl C})$ .



**Figure 1**

The molecular structure of (I), with 30% probability displacement ellipsoids.

### (*E*)-2-[(4-Chloro-1,3-dimethyl-1*H*-pyrazol-5-yl)methyleneamino]benzamide

#### Crystal data

C<sub>13</sub>H<sub>13</sub>ClN<sub>4</sub>O

$M_r = 276.72$

Monoclinic, *C*2/*c*

Hall symbol: -*C* 2yc

$a = 22.046 (3) \text{ \AA}$

$b = 8.6785 (10) \text{ \AA}$

$c = 14.1137 (16) \text{ \AA}$   
 $\beta = 96.005 (2)^\circ$   
 $V = 2685.5 (5) \text{ \AA}^3$   
 $Z = 8$   
 $F(000) = 1152$   
 $D_x = 1.369 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 4144 reflections  
 $\theta = 2.2\text{--}30.2^\circ$   
 $\mu = 0.28 \text{ mm}^{-1}$   
 $T = 296 \text{ K}$   
 Block, colourless  
 $0.32 \times 0.30 \times 0.28 \text{ mm}$

*Data collection*

Bruker SMART CCD  
 diffractometer  
 Radiation source: fine-focus sealed tube  
 Graphite monochromator  
 $\omega$  scans  
 Absorption correction: multi-scan  
 (SADABS; Bruker, 2001)  
 $T_{\min} = 0.725$ ,  $T_{\max} = 1.000$

6634 measured reflections  
 2375 independent reflections  
 2098 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.018$   
 $\theta_{\max} = 25.0^\circ$ ,  $\theta_{\min} = 1.9^\circ$   
 $h = -25 \rightarrow 26$   
 $k = -6 \rightarrow 10$   
 $l = -16 \rightarrow 16$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.035$   
 $wR(F^2) = 0.098$   
 $S = 1.06$   
 2375 reflections  
 175 parameters  
 0 restraints  
 Primary atom site location: structure-invariant  
 direct methods  
 Secondary atom site location: difference Fourier  
 map

Hydrogen site location: inferred from  
 neighbouring sites  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 1.4091P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.17 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.19 \text{ e \AA}^{-3}$   
 Extinction correction: SHELXL97 (Sheldrick,  
 2008),  $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$   
 Extinction coefficient: 0.0266 (12)

*Special details*

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.45129 (2)	0.49955 (4)	0.12551 (4)	0.0612 (2)
N4	0.29251 (6)	0.43146 (17)	0.04310 (11)	0.0531 (4)
H4A	0.3006	0.3480	0.0140	0.064*
H4B	0.3217	0.4899	0.0670	0.064*
O1	0.19254 (5)	0.38809 (14)	0.01737 (10)	0.0600 (4)
N1	0.52045 (6)	0.91074 (16)	0.12334 (10)	0.0486 (4)
N2	0.46079 (6)	0.94242 (16)	0.12143 (10)	0.0445 (3)
C6	0.36052 (7)	0.82150 (17)	0.12489 (11)	0.0397 (4)
H6	0.3417	0.9174	0.1202	0.048*
N3	0.32795 (6)	0.70280 (14)	0.13302 (9)	0.0376 (3)
C1	0.46698 (7)	0.69287 (17)	0.12368 (11)	0.0400 (4)
C2	0.52480 (7)	0.75722 (19)	0.12428 (11)	0.0430 (4)

C3	0.58468 (8)	0.6779 (2)	0.12766 (15)	0.0576 (5)
H3A	0.6159	0.7518	0.1181	0.086*
H3B	0.5831	0.6013	0.0784	0.086*
H3C	0.5938	0.6294	0.1886	0.086*
C4	0.44200 (9)	1.1028 (2)	0.12241 (16)	0.0614 (5)
H4C	0.4316	1.1292	0.1847	0.092*
H4D	0.4072	1.1179	0.0766	0.092*
H4E	0.4749	1.1672	0.1066	0.092*
C5	0.42578 (7)	0.81350 (17)	0.12263 (11)	0.0379 (4)
C7	0.26516 (7)	0.72357 (17)	0.14214 (10)	0.0364 (3)
C8	0.22185 (7)	0.61479 (17)	0.10333 (10)	0.0363 (3)
C9	0.16068 (7)	0.6395 (2)	0.11507 (12)	0.0445 (4)
H9	0.1317	0.5694	0.0890	0.053*
C10	0.14195 (8)	0.7653 (2)	0.16439 (13)	0.0541 (5)
H10	0.1007	0.7807	0.1701	0.065*
C11	0.18475 (8)	0.8675 (2)	0.20506 (14)	0.0577 (5)
H11	0.1726	0.9509	0.2398	0.069*
C12	0.24531 (8)	0.8468 (2)	0.19446 (13)	0.0504 (4)
H12	0.2738	0.9163	0.2227	0.060*
C13	0.23540 (7)	0.46978 (18)	0.05100 (11)	0.0401 (4)

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C11	0.0557 (3)	0.0314 (3)	0.0956 (4)	-0.00049 (17)	0.0037 (2)	-0.0028 (2)
N4	0.0376 (7)	0.0445 (8)	0.0774 (10)	-0.0038 (6)	0.0064 (7)	-0.0244 (7)
O1	0.0391 (6)	0.0426 (7)	0.0960 (10)	-0.0043 (5)	-0.0039 (6)	-0.0204 (6)
N1	0.0387 (7)	0.0446 (8)	0.0618 (9)	-0.0076 (6)	0.0021 (6)	0.0017 (6)
N2	0.0407 (7)	0.0328 (7)	0.0589 (8)	-0.0046 (5)	-0.0004 (6)	0.0008 (6)
C6	0.0404 (8)	0.0316 (8)	0.0452 (8)	0.0002 (6)	-0.0044 (6)	0.0003 (6)
N3	0.0356 (7)	0.0345 (7)	0.0418 (7)	-0.0026 (5)	-0.0006 (5)	-0.0032 (5)
C1	0.0417 (8)	0.0332 (8)	0.0442 (8)	-0.0017 (6)	-0.0001 (6)	-0.0015 (6)
C2	0.0403 (8)	0.0434 (9)	0.0450 (9)	-0.0012 (7)	0.0025 (7)	-0.0004 (7)
C3	0.0447 (10)	0.0609 (12)	0.0674 (12)	0.0047 (8)	0.0066 (8)	0.0000 (9)
C4	0.0561 (11)	0.0322 (9)	0.0946 (15)	-0.0052 (8)	0.0017 (10)	-0.0010 (9)
C5	0.0389 (8)	0.0322 (8)	0.0414 (8)	-0.0043 (6)	-0.0018 (6)	-0.0007 (6)
C7	0.0374 (8)	0.0346 (8)	0.0364 (7)	0.0027 (6)	0.0004 (6)	0.0009 (6)
C8	0.0367 (8)	0.0338 (8)	0.0381 (8)	-0.0006 (6)	0.0018 (6)	0.0023 (6)
C9	0.0366 (8)	0.0452 (9)	0.0513 (9)	-0.0019 (7)	0.0024 (7)	0.0001 (7)
C10	0.0404 (9)	0.0613 (11)	0.0614 (11)	0.0102 (8)	0.0097 (8)	-0.0044 (9)
C11	0.0547 (11)	0.0574 (11)	0.0608 (11)	0.0142 (9)	0.0046 (8)	-0.0175 (9)
C12	0.0478 (10)	0.0461 (9)	0.0557 (10)	0.0014 (8)	-0.0012 (8)	-0.0145 (8)
C13	0.0379 (8)	0.0343 (8)	0.0472 (8)	-0.0027 (6)	0.0009 (6)	-0.0010 (6)

*Geometric parameters (Å, °)*

C11—C1	1.7137 (16)	C3—H3B	0.9600
N4—C13	1.318 (2)	C3—H3C	0.9600

N4—H4A	0.8600	C4—H4C	0.9600
N4—H4B	0.8600	C4—H4D	0.9600
O1—C13	1.2356 (18)	C4—H4E	0.9600
N1—C2	1.336 (2)	C7—C12	1.396 (2)
N1—N2	1.3413 (19)	C7—C8	1.412 (2)
N2—C5	1.360 (2)	C8—C9	1.392 (2)
N2—C4	1.452 (2)	C8—C13	1.505 (2)
C6—N3	1.2677 (19)	C9—C10	1.381 (2)
C6—C5	1.444 (2)	C9—H9	0.9300
C6—H6	0.9300	C10—C11	1.376 (3)
N3—C7	1.4151 (19)	C10—H10	0.9300
C1—C5	1.385 (2)	C11—C12	1.371 (2)
C1—C2	1.391 (2)	C11—H11	0.9300
C2—C3	1.485 (2)	C12—H12	0.9300
C3—H3A	0.9600		
C13—N4—H4A	120.0	H4C—C4—H4E	109.5
C13—N4—H4B	120.0	H4D—C4—H4E	109.5
H4A—N4—H4B	120.0	N2—C5—C1	104.45 (14)
C2—N1—N2	105.89 (13)	N2—C5—C6	121.91 (13)
N1—N2—C5	112.81 (13)	C1—C5—C6	133.62 (14)
N1—N2—C4	118.44 (13)	C12—C7—C8	118.74 (14)
C5—N2—C4	128.70 (14)	C12—C7—N3	120.63 (13)
N3—C6—C5	122.55 (14)	C8—C7—N3	120.52 (13)
N3—C6—H6	118.7	C9—C8—C7	118.35 (14)
C5—C6—H6	118.7	C9—C8—C13	115.57 (13)
C6—N3—C7	118.26 (13)	C7—C8—C13	126.07 (13)
C5—C1—C2	107.22 (14)	C10—C9—C8	121.76 (16)
C5—C1—C11	127.38 (12)	C10—C9—H9	119.1
C2—C1—C11	125.40 (13)	C8—C9—H9	119.1
N1—C2—C1	109.62 (14)	C11—C10—C9	119.45 (16)
N1—C2—C3	121.67 (15)	C11—C10—H10	120.3
C1—C2—C3	128.70 (16)	C9—C10—H10	120.3
C2—C3—H3A	109.5	C12—C11—C10	120.20 (16)
C2—C3—H3B	109.5	C12—C11—H11	119.9
H3A—C3—H3B	109.5	C10—C11—H11	119.9
C2—C3—H3C	109.5	C11—C12—C7	121.41 (16)
H3A—C3—H3C	109.5	C11—C12—H12	119.3
H3B—C3—H3C	109.5	C7—C12—H12	119.3
N2—C4—H4C	109.5	O1—C13—N4	121.44 (15)
N2—C4—H4D	109.5	O1—C13—C8	119.04 (14)
H4C—C4—H4D	109.5	N4—C13—C8	119.52 (13)
N2—C4—H4E	109.5		
C2—N1—N2—C5	0.99 (18)	N3—C6—C5—C1	3.3 (3)
C2—N1—N2—C4	178.50 (16)	C6—N3—C7—C12	-37.2 (2)
C5—C6—N3—C7	175.07 (13)	C6—N3—C7—C8	146.70 (15)
N2—N1—C2—C1	-0.46 (18)	C12—C7—C8—C9	3.1 (2)

N2—N1—C2—C3	-179.23 (15)	N3—C7—C8—C9	179.31 (13)
C5—C1—C2—N1	-0.19 (18)	C12—C7—C8—C13	-175.57 (15)
C11—C1—C2—N1	-179.43 (12)	N3—C7—C8—C13	0.6 (2)
C5—C1—C2—C3	178.46 (17)	C7—C8—C9—C10	-0.9 (2)
C11—C1—C2—C3	-0.8 (3)	C13—C8—C9—C10	177.86 (15)
N1—N2—C5—C1	-1.09 (18)	C8—C9—C10—C11	-1.5 (3)
C4—N2—C5—C1	-178.29 (17)	C9—C10—C11—C12	1.7 (3)
N1—N2—C5—C6	177.42 (13)	C10—C11—C12—C7	0.5 (3)
C4—N2—C5—C6	0.2 (3)	C8—C7—C12—C11	-2.9 (3)
C2—C1—C5—N2	0.75 (17)	N3—C7—C12—C11	-179.15 (16)
C11—C1—C5—N2	179.96 (12)	C9—C8—C13—O1	4.2 (2)
C2—C1—C5—C6	-177.51 (16)	C7—C8—C13—O1	-177.06 (15)
C11—C1—C5—C6	1.7 (3)	C9—C8—C13—N4	-174.85 (15)
N3—C6—C5—N2	-174.72 (15)	C7—C8—C13—N4	3.9 (2)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N4—H4 <i>B</i> ···N3	0.86	2.07	2.749 (2)	136
N4—H4 <i>A</i> ···O1 <sup>i</sup>	0.86	2.10	2.930 (2)	161

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